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Physicochemical Analyses of Selected Clays in Southwestern Nigeria and Their Suitability for Ceramic Production

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Abstract— Clay is the most common ceramic material in the world, which is available in every community. Importance of clay made its enquiries prevalent in agricultural, natural and medical sciences, engineering and ceramics. However, inadequate scholarly attentions on physical and chemical examination of clays that are commonly used for pottery in Southwestern Nigeria necessitate this study. This study is on physicochemical analyses of selected clays in Southwestern Nigeria with the aim to observe their potentialities for ceramics production. Direct field survey, laboratory experimentation and studio art practice methodologies were adopted. Ten clay samples were randomly selected and collected at depth of one metre from two actively used clay depots by potters in Ekiti, Ogun, Ondo, Osun and Oyo States. Samples were sundried, pulverised before their chemical components were analysed with Particle Induced X-Ray Emission (PIXE) technique. Water of plasticity, workability, shrinkage and water absorption capacity of the samples were also examined. Findings revealed that oxide concentrations in ten clay samples vary with high concentration of silicon, alumina and iron oxides range from 27.80%-65.24%, 18.75-43.88% and 1.18%-20.38% respectively alongside other minor and trace elements. Moreover, clays with high concentrations of alumina required more water of plasticity. Secondary clays with high alumina have good plasticity but shrink more than samples with low alumina. Water absorption tests also showed that the samples absorbed less than 20% of their volume which make suitable for ceramic production. However, this study observed that selected clays can be further improved to formulate various clay bodies.

Keywords— physicochemical, clay, Southwestern Nigeria, suitability, pottery, ceramics

I. INTRODUCTION

Clay is formed from the continuous disintegration of the feldspathic rocks such as igneous and granite aided by weathering, transportation and sedimentation of a long period of years. It is a type of mineral that consists mainly of alumina, silica and chemically combined hygroscopic water. No clay is totally pure in nature; hence, chemical composition of ideal clay is Al_2O_3-$2S_1O_2$-$2H_2O$ [1,2]. The types, colours, and plasticity of clay depend on its formation, location and amount of alumina, silica, hydrogen and other oxides that are present in it. Ordinarily, clay is not workable in its raw state until it is subjected to physical pressures. It also becomes more chemically and physically active when adjusted with other clay bodies as well as organic and inorganic materials for specific function. Physical and chemical reactions of clay continue throughout drying stages and when fired to certain degree of [1,3,4}.

There are two types of clay based on geological formation. They are primary and secondary clays. Primary clay is residual or in-situ clay found at the base of mother rock that formed it. It is almost pure in composition because it has little interaction with other foreign bodies which may affect its chemical and physical composition. Primary clay which is also known as kaolin is highly refractory and matures at temperature of about 1400^{o}C and above. It is hardly ever used alone since it has poor plasticity but usually mix with other clays. However, it is the major ingredient of the porcelain body. It is also useful in formulating ceramic bodies to reduce shrinkage and to increase workability and strength of clays [1,4].

Secondary clays are clays that had been moved away from the formation site of the mother rock by wind, erosion and other agents of weathering across space and time. They are more plastic, contain more impurities and varied in colours due to the presence of organic and inorganic materials which mingled with it. Secondary clays are commonly used for ceramic productions because of its high plasticity and availability in large quantity along streams, river banks and valleys. 1,2,4,5].

In Southwestern Nigeria which lies between longitude 2° 30' and 6° East and latitude 6° and 9° North, clay is found abundantly in nearly everywhere. Primary clay (kaolin) is found in few places across the zone while secondary clay is available in every community. However, most of the identified clay deposits are partially exploited while some are yet to be exploited and quantified. Chemical properties and physical potentiality of most of these clays that are used for various ceramic works in the zone have not been scientifically tested [4,6,7,8].

Importance of clay to some agricultural activities, industrial applications and human health made its studies more apparent in Agricultural, Medical and Natural Sciences, Engineering and Ceramics across the globe. Clays are observed to interact relatively with great amount of heavy metals especially in high polluted sediments than the less contaminated deposits and thus useful for absorption of heavy metals in order to recycle industrial waste [9, 10]. Mineralogy, chemical and physical properties of clays from some part of Nigeria has also been examined by various scholars [11,12,13,14,15]. However, there is still in adequate scholastic attention on chemical and physical characteristics pottery clays of Southwestern Nigeria and their suitability for ceramic production. It is against the foregoing that the study on physicochemical analysis of selected clays of Southwestern Nigeria is imperative.

This study is a laboratory experimentation of chemical properties and studio art practice examination of physical characteristics of selected clays for ceramic production. It covers ten clay deposits that are commonly used by the by traditional potters, cottage pottery industries and students tertiary institution that offer Ceramics courses in the Southwestern Nigeria. The aim of this paper is to examine the physicochemical characteristics of selected clays of Southwestern Nigeria with a view to observe their suitability for ceramic production. The objectives of the study are to analyse chemical properties of clay samples, conduct various studio empirical tests on physical properties and observe their appropriateness for ceramic production in order to increase production in Nigeria.

II. Materials and Methods

This study adopted direct field survey, particularly observation, laboratory analysis and studio art empirical examination. Purposive and randomly sampling techniques were used to collect ten clay samples from seventy identifiable clay deposits that were actively used by potters in Southwestern Nigeria. Clay samples were collected from the depth of 1 meter in Isan Ekiti and Igbara Odo in Ekiti State; Igbanran and Abeokuta in Ogun State; Akure and Erusu in Ondo State; Atamora and Titibi in Osun State; Lanlate and Orile-Igbon in Oyo State. No clay deposit was selected in Lagos State as most of identifiable clay sites are now built spaces and for this reason potters in the State usually bought clays that were collected from neighbouring States. Global Positioning System through Goggle Widget on an android phone was used to identify the latitude and longitude of each selected clay site (figure 1 and table 1). Chemical analysis, plasticity, shrinkage and water absorption capacity/porosity test of selected clay samples were further conducted while available scholarly publications were also consulted.

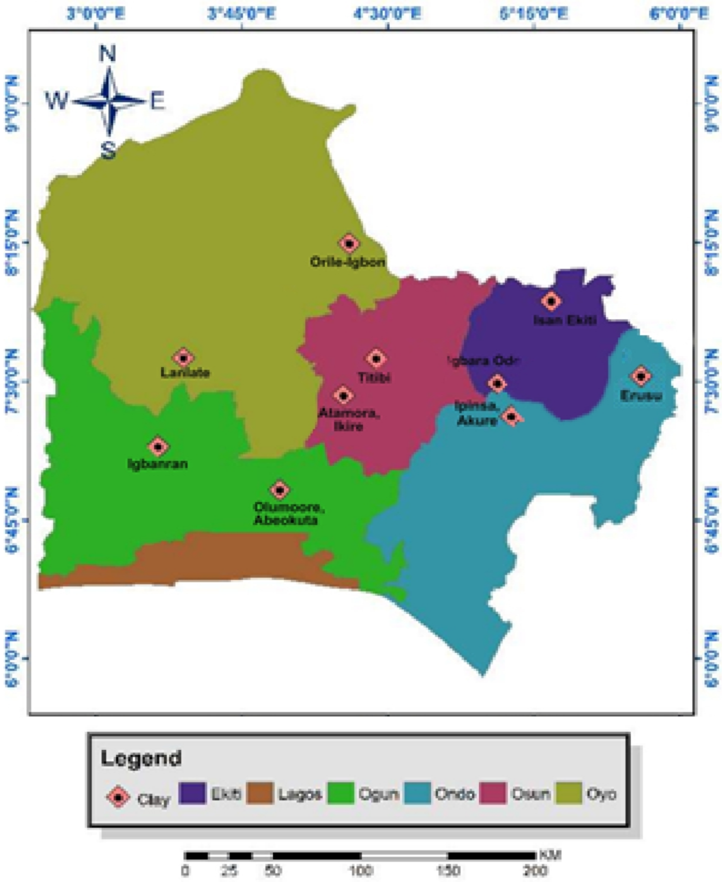

Figure 1: Select Clay Deposits in Southwestern Nigeria Source:

Segun Abiodun, 2021

Table 1: Coordinates of Selected Clay Deposits in Southwestern Nigeria

State	Clay Deposit	Latitude	Longitude
Ekiti	Isan Ekiti	7.9341°	5.33857°
	Igbara Odo	7.489131°	5.063677°
Ogun	Igbanran	6.913118°	3.94474°
	Olumoore, Abeokuta	7.147318°	3.319068°
Ondo	Ipinsa, Akure	7.311225°	5.134608°
	Erusu	7.538594°	5.809892°
Osun	Atamora, Ikire	7.424163°	4.270763°
	Titibi	7.624484°	4.438811°
Oyo	Lanlate	7.627395°	3.44939°
	Orile-Igbon	8.24553°	4.301515°

Source: Authors' Field Study

Chemical Analysis

Collected clay samples were sundried, crushed and then sieved in the ceramics studio. 10g of each of the clay samples were packaged in petri dishes for elemental analysis at Centre for Research and Development, Obafemi Awolowo University, Ile-Ife. Clay samples were further pulverized with Rocklabs CRC3E pulverize machine and pressed into1mm thick and 13mm diameter pellets without binder with hydraulic machine and spec-caps. Elemental properties of clay samples were examined with Particle Induced X-ray Emission (PIXE) of Ion Beam Analysis (IBA). The pelletted clay samples were placed on computer controlled sample holder in the control panel of NEC RC 43 end station in order to scan all the twenty samples. PIXE analysis was done on NEC 5SDH 1.7 MV Pelletron Accelerator by using a 2.5 MeV H+ proton beam. 4mm diametre beam spot and a low current of 3-6Na were used to carried out the measurement from accelerator to the end station with irradiation for about minutes. Data were captured with 4 MCA data collection card on the computer.

Physical Evaluation of Selected Clay Samples

Presence of organic and inorganic materials in clay which caused differences in their chemical compositions also brought about variation of physical characteristics of clays. This section therefore examines plasticity, shrinkage and water absorption capacity/porosity test of selected clay samples so as to observe their suitability of sample for pottery and ceramics activities.

Water of Plasticity and Workability Test

In testing for water of plasticity of selected clay samples, 1kg of each sieved clay sample was measured with digital scale and then poured on a sheet of glass. 500ml of water was measured in a calibrated beaker; the water was gradually added to each clay sample and mixed together simultaneously, wedged until a good texture of workable clay was achieved. Remaining water inside the beaker was then measured to determine the actual amount of water used to mix each clay sample. The weight of each plastic (wet) clay sample was measured and recorded accordingly. This process was repeated thrice for each clay sample while the averages were taken. Percentage of water of plasticity for each sample was calculated with the formula below:

$$\%\textit{Water of Plasticity} = \frac{\text{Weight of Plastic Sample} - \text{Weight of Dry Sample}}{\textit{Weight of Dry Sample}} \times \frac{100}{1}$$

Plasticity of clay samples were also observed through subjective evaluation by rolling kneaded clay into coil of about 3mm diameter and then tied in a knot to examine if it would cracked or not (plates 1 and 2).

Plate 1: Rolling of wet clay into long coil in order to test its plasticity. Photography by Segun Abiodun

Plate 2: Suspending of knoted clay coils in order to test its plasticity. Photography by Segun Abiodun

Slab, coil, throwing and mould casting techniques were also employed to determine workability of each clay samples. Clays were prepared by kneading into balls and then moulded two identical cylinders from each clay sample with slab, coil and throwing methods (plates 3 and 4). Bricks were also casted from 25x10x7cm mould (plates 5)

Plate 3: Moulding of cylinder with handbuilt method. Photography by Segun Abiodun

Plate 4: Forming a cylinder on the wheel. Photography by Segun Abiodun

Plate 5: Casting of bricks in a metal mould. Photography by Segun Abiodun

Shrinkage Tests

Shrinkage of clay is the variation in length of wet, dried and fired ceramic ware due to gradual evaporation of water of plasticity and chemically combined water during drying firing respectively. For this exercise, four clay tiles were produced from each clay sample by pressing well-kneaded clay into 12cm by 12cm wooden halved joints slab making frame place on a flat board (plates 6 and 7). Shrinkages were then examined vis-à-vis linear drying shrinkage, linear fired shrinkage and linear total shrinkage.

Plate 6: Assembling of wooden halved joints slab making frame on a paper. Photography by Segun Abiodun

Plate 7: Pressing of clay sample into the frame. Photography by Segun Abiodun

Linear Drying Shrinkage

Produced tiles were dried in room temperature, turned frequently at interval of seven days to avoid warping. Dried tiles were measured with vernier caliper and drying shrinkage percentage of each tile was calculated with equation below.

$$\%\text{ Linear Drying Shrinkage} = \frac{Wet\ Length - Dried\ Length}{Wet\ Length} \times \frac{100}{1}$$

Linear Fired Shrinkage

Dried tiles that were produced were arranged in updraught gas kiln and fired to Orton cone 09, that is, 928°C. Each fired tile was also measured and recorded in order to calculate the percentage of linear fired shrinkage in the equation 2 below:

$$\%\text{ Linear Fired Shrinkage} = \frac{Dried\ Length - Fired\ Length}{Dried\ Length} \times \frac{100}{1}$$

Linear Total Shrinkage

Linear total shrinkage is the overall shrinkage of clay which shows differences in size of wet and fired ceramic tiles. Percentage of linear linear Total Shrinkage was calculated with the formula below:

$$\%\text{ Linear Total Shrinkage} = \frac{Wet\ Length - Fired\ Length}{Wet\ Length} \times \frac{100}{1}$$

Water Absorption Capacity Test

Water absorption capacity exanimations were also conducted to observe the amount of water that each clay tile would absorb when immersed. Water absorption techniques by soaking clay tiles in cold water for 30 minutes, soaking tiles in cold water for 24 hours and boiling clay tiles on fire for 5 hours with 19 hours cooling were adopted for both sieved and unseived clay samples (plates 8 and 9). Boiling clay samples on fire for 5 hours is in accordance to BS 3921:1985 - British Standard specification for clay brick while soaking in cold water for 24 hours is for quality control [16]. Saturated tiles were removed; excess water was cleaned from the surface and then weighing on scale. Percentages of absorption capacity of clay tiles were calculated in equation below.

$$\%\text{ Water Absorption Capacity} = \frac{Soaked\ Weight - Fired\ Weight}{Fired\ Weight} \times \frac{100}{1}$$

Plate 8: Clay tiles were soaked in cold water for 24 hours. Photography by Segun Abiodun

Plate 9: Boiling of clay tiles on electric stove for 5 hours. Photography by Segun Abiodun

III. Results And Discussion

Chemical Analysis

Elemental concentrations of selected clay samples were given in part per million (ppm) which were later converted to percentage with the formula below.

$$\% \text{ Elemental Concentration} = \frac{\textit{Elemental Concentration in ppm}}{1{,}000{,}000} \times \frac{100}{1}$$

Each elemental concentration was further converted into oxide since elements in clay are chemically combined with hydrogen and oxygen; that is water (H_2O) during formation. This was done through the aid of the formula below. The oxide concentrations of the selected clay samples were therefore given in table 2 below:

$$\% \text{ Oxide Concentration} = \frac{\% \textit{ Elemental Concentration} \times \textit{Molecular Weight}}{\textit{Atomic Weight}}$$

Table 2: Oxide Concentrations of Selected Clay Samples in Southwestern Nigeria

State	Clay Deposit	Oxide Concentration (%)											
		MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	Ti_2O	MnO	Fe_2O_3	ZnO	Rb_2O	SrO
Ekiti	Isan Ekiti	1.35	18.75	27.80	0.11	1.30	38.87	3.87	0.06	8.18	0.02	0.01	-
	Igbara Odo	0.89	28.08	55.65	0.07	3.25	0.81	1.37	0.08	9.74	0.03	-	0.03
Ogun	Igbanran	1.88	32.28	46.24	0.15	0.45	2.46	3.02	0.14	13.01	0.07	-	0.01
	Olumoore, Abeokuta	1.17	20.68	65.24	0.29	2.39	0.69	2.11	0.24	7.15	-	0.01	0.02
Ondo	Ipinsa, Akure	0.08	43.73	48.76	0.90	0.01	0.02	3.37	-	3.93	-	-	-
	Erusu	0.83	27.86	56.38	0.12	1.49	0.92	1.44	0.04	10.87	0.03	-	-
Osun	Atamora, Ikire	3.70	30.05	48.96	0.09	2.88	1.06	1.42	0.04	12.14	-	0.04	0.05
	Titibi	0.27	43.88	52.67	0.08	1.66	0.08	0.02	0.01	1.18	-	0.06	-
Oyo	Lanlate	1.73	35.22	52.69	0.07	0.93	2.39	0.66	0.04	6.34	-	-	0.03
	Orile-Igbon	0.98	31.46	44.18	0.04	0.89	0.49	1.50	0.03	20.38	0.02	0.01	0.01

Source: Authors' Laboratory Analysis

From the table 3 above, silicon (Si) and aluminium (Al) which are the basic elements of ideal clay were present as the major elements in all selected samples. Iron (Fe) was found as a major element in eight (80%) of the secondary clay samples and trace element in two (20%) primary clay samples. Potassium (K), an alkaline flux in clay was found in low concentration in all the selected clay samples and it is present in five (50%) samples as major element, in four (40%) as minor element and in one (10%) as trace element. Titanium (Ti) was present in seven clays (70%) clay samples as major element, in two (20%) as minor elements and trace element in one (10%) of the samples. Magnesium (Mg) was found as a major element (though low) in five (50%) of the clay samples, minor element in four (40%) clays and trace element in one (10%) of the samples. Calcium (Ca) was also present as major element in four (40%) clays, minor element in four (40%) samples and trace element in two (20%) samples.

Phosphorus (P) was found in five (50%) clay samples as minor element and in other five (50%) samples as trace element. Manganese (Mn) was found in nine (90%) clay samples as it present in one (10%) sample as minor element and other eight 80%) samples as trace element. Each of the Strontium (Sr), Zinc (Zn) and rubidium (Rb) was also present in five clay (50%) samples as trace element.

Water of Plasticity and Workability

The results percentage of water of plasticity that each clay sample requires to be workable is given in the table 3 below.

Table 3: Water of Plasticity of Sieved Selected Clay Samples in Southwestern Nigeria

State	**Clay Deposit**	**Dry**	**Volume**	**Wet**	**Water of Plasticity**	**% Water of Plasticity**	**Plasticity**
Ekiti	Isan Ekiti	1kg	310ml	1.295kg	0.295kg	29.5%	Good
	Igbara Odo	1kg	300ml	1.255kg	0.255kg	25.5%	Good
Ogun	Igbanran	1kg	300ml	1.270kg	0.270kg	27.0%	Good
	Olumoore, Abeokuta	1kg	285ml	1.250kg	0.250kg	25.0%	Good
Ondo	Ipinsa, Akure	1kg	570ml	1.550kg	0.550kg	55.0%	Poor
	Erusu	1kg	275ml	1.245kg	0.245kg	24.5%	Good
Osun	Atamora, Ikire	1kg	270ml	1.245kg	0.245kg	24.5%	Good
	Titibi	1kg	420ml	1.440kg	0. 440kg	44.0%	Poor
Oyo	Lanlate	1kg	300ml	1.27kg	0.27kg	27%	Good
	Orile-Igbon	1kg	380ml	1.385kg	0.315kg	31.5%	Good

Source: Authors' Studio Test

Clay samples with high concentrations of alumina and fine particles after sieving absorbed more water than samples with low concentrations of alumina. Though clay samples from Ipinsa and Titibi required more water to be plastic but their plasticity is poor because they were in-situ clays which usually have poor plasticity because of their little or no interaction with organic materials.

On their workability, earthenware clay samples from Isan Ekiti; Igbara Odo; Igbanran; Olumoore, Abeokuta; Erusu; Atamora;; Lanlate and Orile-Igbon are suitable for moulding of ceramic wares with various handbuilt, throwing and mould casting techniques because of their good plasticity. Kaolin samples from Ipinsa in Akure and Titibi alone are not good for ceramics production because the wares that were produced from these samples were deforming during and after production because of their low plasticity and unmalleability (plate 10). However, addition of little plastic clays to kaolin samples from Ipinsa and Titibi will improve their plasticity and workability. Other earthenware clay samples under this study can be solely used for ceramic production, though addition of non-plastic bodies suck as kaolin, grog, sawdust, laterite, waste paper to them will yield better results for various purposes.

Plate 10: Deformed dried cylinders made from Ipinsa in Akure, and Titibi clay samples. Photography by Segun Abiodun

Shrinkage Tests

The results the three shrinkage tests are shown in table 4 below.

Table 4: Shrinkage Analysis of Selected Clays in Southwestern Nigeria

State	Clay Deposit	Plastic Length	Shrinkages					
			Drying Length	% Linear Drying Shrinkage	Firing Length	% Linear Firing Shrinkage	Linear Total Shrinkage	% Linear Total Shrinkage
Ekiti	Isan Ekiti	12cm	11.10cm	7.5%	10.65cm	4.05%	1.35cm	11.25%
	Igbara Odo	12cm	11.18cm	6.88%	10.95cm	2.01%	1.05cm	8.75%
Ogun	Igbanran	12cm	11.25cm	6.25%	10.8cm	4.0%	1.2cm	10.0%
	Olumoore, Abeokuta	12cm	11.10cm	7.5%	10.65cm	4.05%	1.35cm	11.25%
Ondo	Ipinsa, Akure	12cm	11.48cm	4.33%	11.25cm	1.96%	0.75cm	6.23%
	Erusu	12cm	11.03cm	8.11%	10.73cm	2.72%	1.28cm	10.67%
Osun	Atamora, Ikire	12cm	11.18cm	6.83%	10.8cm	3.36%	1.2cm	10.0%
	Titibi	12cm	11.55cm	3.75%	11.25cm	2.60%	0.75cm	6.25%
Oyo	Lanlate	12cm	10.8cm	10%	10.35cm	4.17%	1.65cm	13.75%
	Orile-Igbon	12cm	10.8cm	10%	10.5cm	2.78%	1.5cm	12.5%

Source: Authors, Studio Test

Percentages of linear drying, linear fired and linear total shrinkages of selected clay samples range from 3.75% - 12.5%; 2.01% - 4.17 and 6.23% to 13.75% respectively. These results show that the selected clay samples are good clays because none of them shrink up to 15% of their linear size and this make suitable for pottery and bricks production. Moreover, clay samples that with high shrinkage can be modified by adding less shrink clay, sand grog or non-plastic material such as paper, sawdust and other agricultural wastes in order to reduce their shrinkages.

Water Absorption Capacity

The result of percentage of water absorption tests by soaking in cold water for 30 minutes and 24 hours; and boiling for 5 hours with 19 hours cooling of both sieved and unsieved selected clay samples were given in table 5 below.

Table 5: Percentages of Water Absorption Capacity of Selected Clay Samples of Southwestern Nigeria by Soaking in Cold Water for 30 Minutes and 24 hours; and Boiling for 5 Hours

State	**Clay Deposit**	**Sieved Clay**			**Unsieved Clay**		
		% of Absorption Capacity in 30 Minutes Soaking	**% of Absorption Capacity in 24 Hours Soaking**	**% of Absorption Capacity in 5 Hours Boiling**	**% of Absorption Capacity in 30mins Soaking**	**% of Absorption Capacity in 24 Hours Soaking**	**% of Absorption Capacity in 5 Hours Boiling**
Ekiti	Isan Ekiti	5.22%	9.57%	11.30%	6.36%	10.9%	12.73%
	Igbara Odo	13.33%	17.5%	19.20%	15.0%	19.17%	21.67%
Ogun	Igbanran	11.67%	14.17%	17.50%	12.31%	15.39%	18.86%
	Olumoore, Abeokuta	14.17%	17.5%	19.2%	14.29%	18.1%	19.05%
Ondo	Ipinsa, Akure	28.42%	31.57%	33.33%	28.42%	31.57%	33.33%
	Erusu	10.4%	13.6%	16.30%	11.2%	15.2%	16.67%
Osun	Atamora, Ikire	10.35%	15.39%	16%	13.08%	16.15%	17.5%
	Titibi	24.55%	28.18%	31.58%	28.33%	30.83%	31.3%
Oyo	Lanlate	9.52%	12.38%	13.64%	10.48%	13.%	15%
	Orile-Igbon	10.48%	13.33%	15.79%	13.33%	15.34%	16.19%

Source: Authors' Studio Test

The results from the table above revealed that all unseived clays absorbed more water than their sieved clays. This is because coarse particles in most of the unseived clay prevent proper close compact of particles in the clay tiles and thereby create cracks on the tiles during firing. Initial suction rate of all the tiles were high but reduced as all the pores were filled up in other two tests. Clay samples from Ipinsa in Akure and Titibi have highest water absorption rates of 28.42 and 33.33% and they were very weak after the tests. This is as a result of being not yet matured at 928^{o}C as they are primary and high temperature clays. Nearly all the secondary clay samples have absorption capacity that is lower than 20% at the both soaking in cold and boiling except for clay from Igbabra Odo that has more than 20% absorption during boiling. These results show that the clay samples have high strength for various ceramic productions because strength and quality of a clay sample is associated to the less water absorption as a good clay is must not to absorb more than 20% water of its weight [16,17].

IV. Conclusion

Chemical and physical properties of selected clay samples revealed their individual usability and suitability in ceramic productions. Studio tests showed that elemental concentrations in clay samples have diverse effects on physical characteristics of clays such as water of plasticity, shrinkage, firing characteristics and water absorption capacity. Water of plasticity test revealed that besides fineness of particle sizes of clay samples, selected clay samples with high concentration of aluminium absorbed high volume of water. Clay samples with high concentrations of aluminium have poor plasticity but they have low shrinkage during drying and firing which make the suitable to reduce the shrinkage of plastic clay during clay body formulations.

Nearly all the secondary clay samples under this studied are good for different pottery production. Clay samples from Ipinsa, Akure; and Titibi which are kaolin cannot be used for modelling alone but they can be mixed with other clays with good plasticity in varying proportions to improve their workability and strength. All clay samples under this study can be further improved upon to compound various clay bodies that are suitable for production of pottery, water filters and bricks and insulators among others.

In conclusion, this study recommends inquiry of basic knowledge in material sciences which will enable potters and ceramists to understand their material beyond empirical understanding for effective pottery production. Potters especially Ceramics Researchers Association of Nigeria (CeRAN) and Craft and Potters Association of Nigeria (CPAN) with various concerned governmental and non-governmental agencies should solely and jointly be engaged in thorough examination of chemical and physical potentialities of various clay samples across the country; thus making the data available for clay users and prospective entrepreneurs in the clay industry in order to increase various ceramic production in Nigeria in order to reduce importation of ceramic wares in the country.

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